



WESTON WAY
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14 November 1989

Mr. Michael Towle
Remedial Project Manager
U.S. EPA
841 Chestnut Building (3HW21)
Philadelphia, PA 19107

Dear Mr. Towle:

Attached are the addendums to the Commodore RISOP. The initial pages is the updated schedule based on the 11 October 1989 RISOP approval date. The second page is an updated sampling locations map to include the residences sampled in August on Apple Valley Lane. Table 1-11 replaces the one in the spiral bound RISOP. It contains corrected reference section locations in addition to the other corrections noted in the EPA comment letter of 18 September 1989.

Page 1 starts the answers or fulfills the comments of the September letter. WESTON trusts that these will satisfy the concerns of EPA. Also please be assured that the RISOP was written with room for adjustment. That is WESTON intends to maintain close and consistent contact with EPA throughout this investigation so that if a particular procedure requires alteration, EPA, Commodore and WESTON will all review the data and the reasoning to make the most positive change possible.

Page ten starts the answers to the QA comments. These were a direct result of WESTON's QA staff contacting Dianne Simms at EPA's CRL again and answering the comments point by point. Again WESTON's intent is to work as close as possible with EPA to insure that all data collected are precise, accurate, complete, representative and comparable.

Should you have any further questions, please call me at 430-3047, Charles Kufs at 430-3049 or Steve Jakatt at 430-7254.

Very truly yours,

ROY F. WESTON, INC.


for Katherine Sheedy, P.G., V.P.
Project Director

KS/rmk

Attachments

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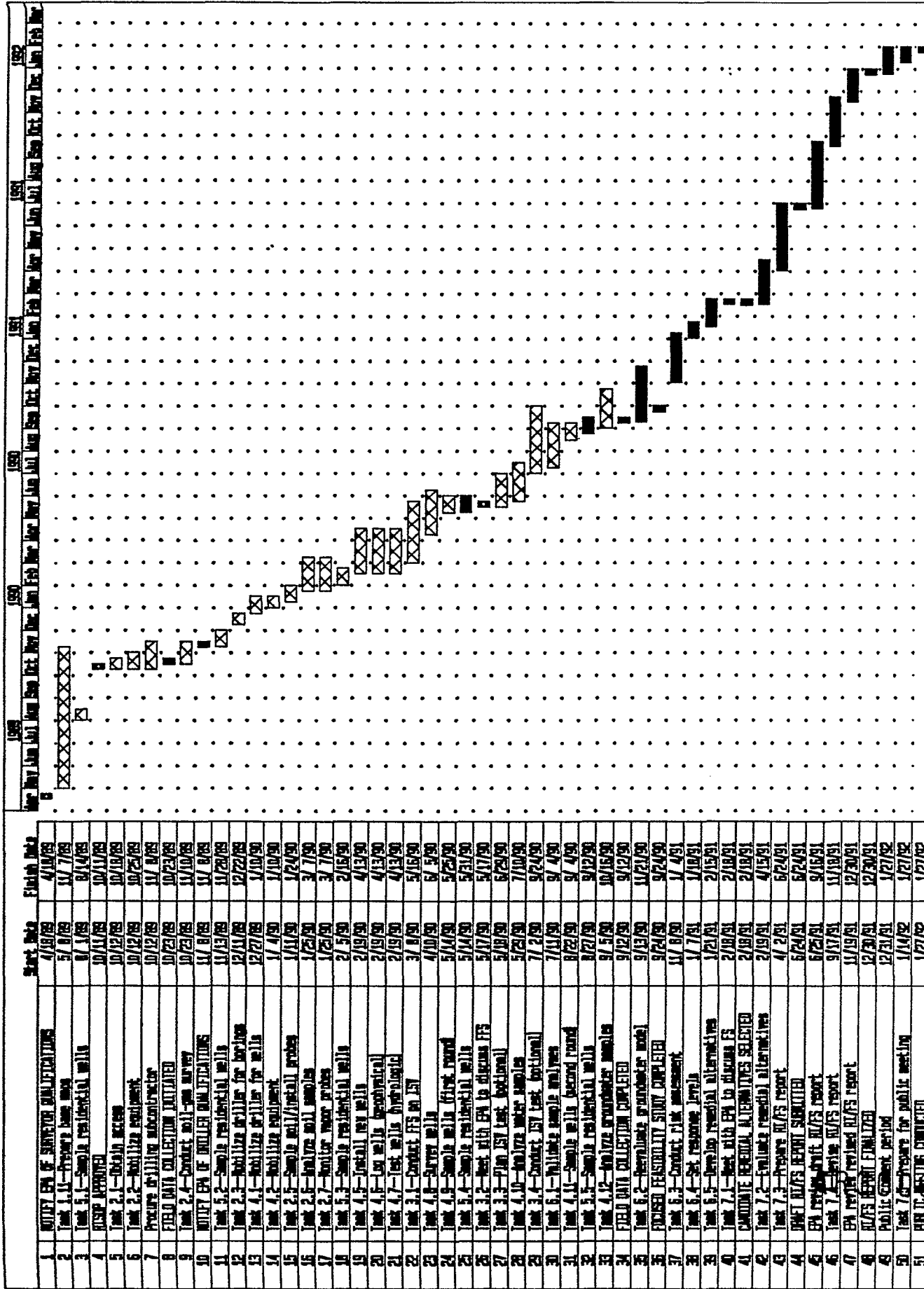
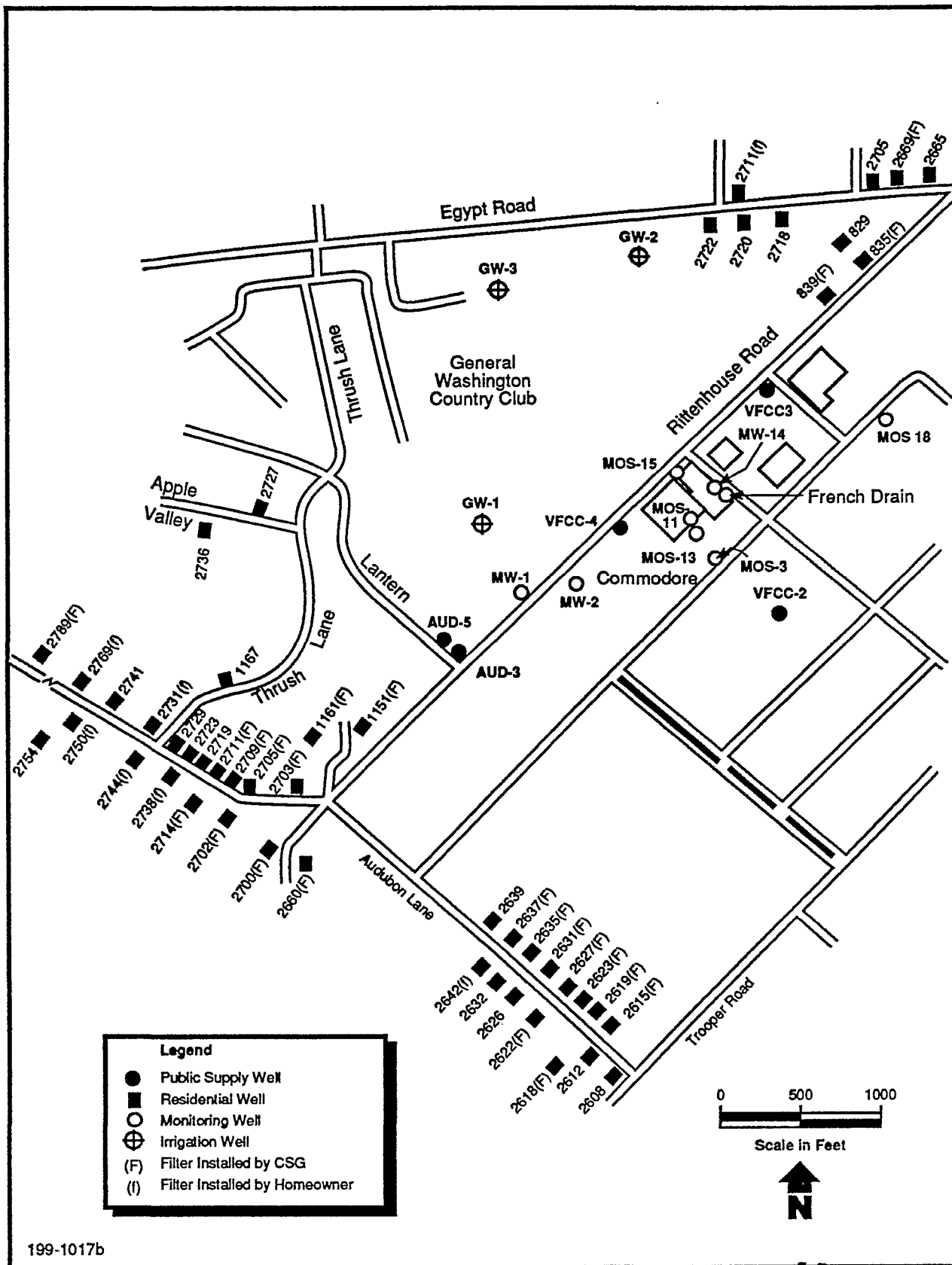


Figure 5-1. Schedule of Tasks for the RI/FS of the CSG Site on Rittenhouse Road



199-1017b

**FIGURE 1 PREVIOUS AND CURRENT SAMPLING LOCATIONS,
VALLEY FORGE CORPORATE CENTER AREA**

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Table 1-11

**Commodore Semiconductor Group
Summary of RI Work Tasks and RISOP Organization**

Task*	Objective	Data Use	Selected Analytical Options	Sensitivity	Analytical Methods	No. of Samples	Duplicates/Splits	RISOP Reference Section	Notes
1.11 Obtain aerial photographs	Obtain up-to-date photo coverage to assess culture and provide base for site and regional base maps.	1, 3	N/A	N/A	N/A	N/A	N/A	1.3.2	Task performed March 1989
1.11 Prepare Base Maps	To aid in planning and implementing the RI	1, 3	N/A	N/A	N/A	N/A	N/A	1.3.2	Task performed August 1989
2.1 Obtain Site Access	To gain permission to sample or install monitoring wells on properties not owned by CSG	1, 3	N/A	N/A	N/A	N/A	N/A	1.3.5	10 potential properties
1.6 Water Level Measurements	Determine depth to water. Measurements used to construct potentiometric surface maps to determine direction of groundwater flow	1, 3, 5	I	N/A	Depth-to-water meter; Steven's recorder; PID	4 quarterly rounds: July 1989 - March 1990; Monthly: April 1990 through October 1990; "Steven's" type recorder on 1 well (MOS-11); March 1990 through Sept. 1990	N/A	1.3.6	1 round equals a maximum of 15 existing wells plus 9 new wells
2.4 Soil-Gas Survey	Evaluate concentration and movement direction of VOCs in overburden to direct placement of future monitoring wells	1, 3, 6	II	Low ppm	Field Lab GC with dual ECD	300	12	2 daily 1.3.7 Lab Standard 2 daily	

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Table 1-11
(continued)

Task	Objective	Data Use	Selected Analytical Options	Sensitivity	Analytical Methods	No. of Samples	Duplicates/Splits	Blanks	RISOP Reference Section	Notes
2.5 Drill Soil Borings and Install Vapor Probes	To classify overburden composition, and allow installation of vapor probes for quarterly vapor monitoring	1, 3, 4, 6	N/A	N/A	N/A	N/A	N/A	N/A	1.3.8	Vapor probes will be monitored during quarterly groundwater sampling program
2.5 Collection of Soil Samples	Visually characterize overburden, scan split-spoon samples for real-time VOC content, and obtain sample at soil/bedrock interface	1, 2, 3, 4	I	Low ppb (VOC)	Field Instrument PID	15 VOC			1.3.9	25% of soil samples collected will be for TCL/TAL analyses
2.6 Analysis of Soil Samples	To detect halogenated volatile organic compounds in soil samples collected; also, 25% of soil samples will be analyzed for possible TCL/TAL compounds	1, 2, 3, 5	I, III, IV	Low ppb (VOC) Low ppb (TCL/TAL)	VOC 8010 8020 TCL/TAL	15 VOC 4 TCL/TAL	1 1	1	1.3.11	25% of soil samples will be analyzed for TCL/TAL compounds
4.4 Installation of Monitoring Wells	To characterize the overburden and bedrock types; to provide information on the direction and rate of groundwater and contaminant movement; provide information for developing a model to design an effective remediation plan	1, 2, 3, 4, 5	I	Low ppb (VOC)	Field Instrument	Cores of deep wells maximum 3	N/A	N/A	1.3.12	Wells will be installed in "clusters" containing 1 deep, 1 shallow bedrock, and 1 overburden well. Three clusters will be installed. A fourth will be installed if data from soil-gas and soil-boring programs indicate the necessity
4.5 Borehole Geophysics	To provide in situ description of bedrock	1, 3, 4	N/A	N/A	GEO-LOG	Up to 4 new wells	N/A	N/A	1.3.13	To include (if possible) AUD MW-1 and 2 also MOS-11, 18

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Table 1-11
(continued)

Task	Objective	Data Use	Selected Analytical Options	Sensitivity	Analytical Methods	No. of Samples	Duplicates/Splits	Blanks	RISOP Reference Section	Notes
2.7 Monitor Vapor Probes	Define the aerial extent of VOC's at soil-bedrock interface	1, 3, 5, 6	I	Low ppm (VOC)	PID	15	N/A	N/A	1.3.14	Monitor monthly April 1990 through October 1990
4.6 Hydrologic Well Testing	To determine zone hydraulic conductivity, zone interconnection. Water analysis to determine VOC concentrations in discrete zones.	1, 3, 4, 5	II, III		TD/RAS	Up to 30 intervals, up to 20 water samples	N/A	N/A	1.3.15	
4.8 Collect Water Samples	Collect groundwater from residential wells, irrigation wells, monitoring wells and, if possible, from public supply wells to support site characterization	1, 2, 3, 4, 6	I	Low ppb (VOC) Low ppb (TCL/TAL)	PID SC pH Temp.	38 to 41 (1) 52 to 55 (2) 2 monitoring wells	3 3	6	1.3.16	1 round of pre-filter/post-filter residential wells. TAL samples to be filtered
4.9 Analyze Water Samples	Detect VOCs in water samples collected at and around the CSG site	1, 2, 3, 4, 5, 6	III IV	RAS CLP	EPA 601/602 TCL/TAL	39 to 42 (1) 53 to 56 (2) 2	3 3	6	1.3.17	
4.7 Survey Wells	To accurately locate all wells in both horizontal and vertical planes	1	N/A	N/A	N/A	N/A	N/A	N/A	1.3.18	Subcontractor services
4.3 Air Emissions Modeling and Testing	Test the downstream effects of the exhaust gas from the air strippers	2, 4, 6	II	CRDL	N/A	2	1		1.3.19	

NOTES:

* See Section 1.3.3 Schedule of Tasks

DATA USE:

1. Site Characterization
2. Risk Assessment
3. Evaluation of Alternatives
4. Engineering Design of Alternatives
5. Contamination Transport Evaluation
6. Health and Site Safety



Attachment to CSG RISOP

Comment 1:

Response: No Action Required

Comment 2A:

Response A: In cooperation with public and EPA request of August 1989, Commodore has already sampled several residential wells on Apple Valley Lane for VOC's in August 1989. Groundwater from those wells was within MCL guidelines set by EPA.

Response B: The sampling program described in this RISOP was verbally approved by representatives of both EPA and PADER. The sampling, as stated on page 1.3-55 specifies sampling at the 11 residential wells that were originally installed with whole-house filters and where at least one groundwater sample has contained compound concentrations above the MCL. Since the August 1989 sampling one more well has been added to this list. Sampling these wells is a priority. The RISOP also makes provisions for including at least five other wells which may or may not have tested positive for any of the chemicals of concern. Each quarterly sampling round has concentrated on acquiring samples from residences where sampling has not been performed for more than three quarterly rounds. At each round, many attempts are made by phone, by mail and by knocking on doors during the day, the evening and on weekends. After many attempts the targeted residence may not be sampled during that specific round; however the residence of concern is given top priority for the next round. Statistically, the number of residences that can be sampled in a seven-day sampling event is approximately 17. All of the original 22 wells with filters, most of the original 43 wells and other random wells like those on Apple Valley are hoped to be sampled during the RI at least once.

The Priority Residences are as follows:

2623 Audubon Road
2660 Audubon Road
2730 Audubon Road
2705 Audubon Road
2709 Audubon Road
2711 Audubon Road
2714 Audubon Road
2669 Egypt Road
835 Rittenhouse Road

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839 Rittenhouse Road
1151 Rittenhouse Road
1161 Rittenhouse Road

The following scenario is adhered to during each round in order of decreasing priority:

<u>Round 1</u>	<u>Round 2</u>	<u>Round 3</u>	<u>Round 4</u>
Planned Wells Priority Res.	Priority Res.	Priority Res.	Priority Res.
Residence-A	Residence-C	Residence-G	Residence-Q
Residence-B	Residence-G	Residence-I	Residence-S
Residence-C	Residence-I	Residence-O	Residence-T
Residence-D	Residence-J	Residence-P	Residence-AA
Residence-E	Residence-K	Residence-Q	Residence-BB
Residence-F	Residence-L	Residence-R	Residence-CC
Residence-G	Residence-M	Residence-S	Residence-DD
Residence-H	Residence-N	Residence-T	Residence-EE
Residence-I	Residence-O	Residence-U	Residence-FF
<u>Not Sampled</u>	<u>Not Sampled</u>	<u>Not Sampled</u>	
C, G, I	G, I, O	Q, S, T	

Comment 3:

Response 3: For an individual to gain access to the residential well analysis he should send a letter of request to:

Commodore Semiconductor Group
950 Rittenhouse Road
Norristown, PA 19403
Attention Mr. Richard Ng

All homeowners or residents have been apprised of this verbally at each sampling.

Comment 4:

The first paragraph of Section 1.3.2 will now read:
To meet the original schedule set by EPA for this RI and to provide an accurate system for locating data points, aerial photographs were taken of the site in early spring of 1989. This was done with prior knowledge and approval of EPA. These aerial photographs were then used to prepare digitized common land base maps. This was also performed with the prior approval of EPA.

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Addition to Appendix B

Appendix BB

Water Level Measurement with Stevens^R - Type Recorder

BB.1 ASSOCIATED PROCEDURES

General Equipment Decontamination (see Appendix N)

BB.2 PREPARATION

BB.2.1 Office

Check meter for proper operating condition. There must be a working cartridge pen and a new replacement available before the meter is brought to the site. There must be adequate chart paper available as well as the necessary timing gears. The Site Manager will determine the proper timing gears required which will depend on the expected water level change. Six new size D batteries will be acquired to power the meter.

BB.2.2 Field

Construct a small platform to support the meter directly over the top center of the well to be measured. The platform must be level and able to support the meter in a stable position for approximately 7 months.

BB.3 OPERATION

- A. Ensure that all parts of the meter that will enter or touch the well are new or thoroughly decontaminated.
- B. Check the platform for horizontal level and structural support.
- C. Attach the chart paper and cartridge pen. Make sure the proper gears are installed for timing and water-level differential.
- D. Measure depth to water. Measure height of meter float pulley above top of well. Cut pulley wire twice the distance from top-of-water to top-of-pulley so the float and counterweight will hang just at the top of water.

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- E. Center meter on support stand so float and counter weight support wires are centered in well. Lower float and counterweight.
- F. Note the time of day on the chart for initiating the record and locate the corresponding time-line with respect to the time scale with which the recorder is equipped.
- G. Note the depth-to-water on the chart for the beginning recording period. Also mark the corresponding position on the chart in accordance with the ratio of pen movement to actual water level change.
- H. Observe the meter for an appropriate period of time to ensure proper working order.
- I. Cover meter to keep the elements from disrupting the recording chart of the mechanism.
- J. Periodically check depth-to-water and write this on the chart paper as well as in the field notebook.

BB.4 POST OPERATION

BB.4.1 Field

- A. Decontaminate the float and counterweight as well as any parts that may have come in contact with the well.

BB.4.2 Office

- A. Give all original charts to the site manager for eventual delivery to CSG and EPA.

BB.5 REFERENCES

Leupold and Stevens, Inc., "Instruction Manual; Publication Part number: 40349, STEVENS Model 68 Type F Recorder with Quartz Multispeed Timer" March 15, 1984.

Comment 6

Add to Appendix K

K.3.b

11.A. Data logger calibration:

Starting System Tape

- 1. Turn the computer OFF.

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2. Verify that the record interlock on the system tape is in the right (secure) position.
3. Insert the system tape in the tape drive.
4. Turn computer ON.
5. Computer will automatically start the test program.

11.B. Troubleshooting

SYMPTOM	PROBLEM	CORRECTIVE ACTION
Computer displays System Communication error note	There are three basic possible problems:	
	1. The Data Acquisition Unit is not turned on.	Turn on DAC
	2. The HP-IB is disconnected from either or both the Data Acquisition Unit or the computer	Turn off system and IB; restore interconnections
	3. AC power to the system has voltage transients.	Use uninterruptible power supply.

11.C. Background Test Operation

The background Test software is designed to measure the water level data at a constant rate.

11.C.1 Preparation for Test. The program accepts the following test parameter specifications:

1. Job number.
2. Run number.
3. Test date and start time.
4. Units of water level measurement.
5. Time units.
6. Test duration.
7. Data sample rate.
8. Number of wells to be measured.
9. Well identification numbers.
10. Transducer data (for transducers in each well)

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- a. Transducer serial number.
- b. Transducer scale factor.
- c. Water level.

11.C.2 Pre-run Status. The program provides a check of water level readings to allow the geologist to validate pre-test conditions.

11.C.3 Real-time Test Data. The program provides real-time water level plots or data tables on any well.

1. Water level in a selected well can be displayed as a plot on the computer screen and this plot can be copied at any time.
2. A data table of water level in a selected well can be printed at any time; the time interval between points held for display or printout during a test is test duration/125 or one minute, whichever is greater.
3. A test status display is available; this presents a table of real-time readings in each well so that all water levels can be viewed simultaneously.
4. A continuous real-time printout of water level in any selected well can be maintained.

11.C.4 Test Completion. The test can be completed using several options.

1. The computer can end the test automatically upon expiration of the test duration specified during preparation.
2. The test can be terminated manually by the operator at any time.
3. The test duration may be extended at any time during the test.

After the test has ended, a final report can be automatically printed, giving test specifications and the following as desired:

1. Plots of water level in each well.
2. Complete data tables of water level in each well.

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11.C.5 Test Software Operation. Use the procedure described in Owner's Manual: Hydrologic Analysis System, Model SE-200 In-Situ Inc., 1982, Revised 4/84, Appendix C for software operation. The Owner's Manual will be available at the site when the SE-200 is being utilized. Part 11C is specifically designed to describe the constant rate test software operation; however, the background test is just a simplified version of the constant rate test.

Add to Appendix K

K.3.b

12.A Transducer Calibration

Using the system tape, set up the transducer as for normal water level measurement. Set up for the pre-run check option (see Operator's Manual, In-Situ, Inc. Appendix C, p. 28) and proceed as follows:

12.B. Pre-run Check

1. Submerge transducer at a water level below the seasonal temperature variation.
2. Accurately measure off and mark a known length on the transducer cable. For maximum accuracy, this length should approximate the transducer range minus the hydrostatic head of the initial transducer setting. For example, if the transducer range is 60 meters and the initial transducer setting submerges the transducer 2 meters below the water level, the measured length should be approximately 58 meters (units of measurement need not be metric).
3. Record the water level indicated by the computer.
4. Lower the transducer the measured distance of step 2 to its second setting.
5. Record the water level indication for this second transducer setting.
6. Calculate the transducer scale factor using the following equation:

$$Sf = Sf1 \times \frac{L}{W} ,$$

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where Sf = new scale factor
 L = length measured in step 2
 W = difference between water level
 indications of steps 3 and 5
 Sf1 = old scale factor.

If the old scale factor (Sf1) is unknown, substitute K from the following table.

Scale Factor Verification

Transducer Range (meters)	K
6	10
30	50
60	100

Note:

The following factors will introduce errors when performing calibration by this technique.

- o Extreme temperature gradients related to shallow water tables, geothermal sites, etc.
- o Thermal expansion and contraction of air within the transducer cable vent tube as the cable passes between warmer and cooler environments outside and inside the well (transient error).
- o Slug effect caused by volumetric displacement of the transducer and cable in a low permeability situation.

12.B.7 PRESSURE TRANSDUCER SPECIFICATIONS

Excitation Voltage	9 to 30 VDC
Output	4 to 20 mA
Setting Accuracies	
Zero	4 mA, +0.5% FS
Full Scale	20 mA, -1% FS

Non-Linearity	±0.1% FS
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Hysteresis	±0.02% FS
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Repeatability	±0.02% FS
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Self Noise	Nominal 0.05% FS Pk to Pk with 2.5 KHz cutoff
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Long Term Stability	0.2% FS per year
Warm Up	Nominally 0.05% FS, 30 to 60 secs
Accuracy	
At Constant Temp:	$\pm 0.1\%$ of Range
Temperature Error Band at Constant Pressure	$\pm 0.3\%$ of range over a temperature range of -2° to 30°C ; 28°F to 86°F
Operating Temperature Range	-20°C to 80°C ; -4°F to 176°F
Storage Temperature Range	-55°C to 95°C ; -67°F to 203°F
Overpressure Capability	2X Full Range
Media Compatibility	Groundwater with dissolved H_2S , seawater, salt brine
Size	0.59" dia. X \approx 8.5" long
Pressure Connection	0.58" x 32 TPI 55 DEG (Witworth form)
Wetted Materials	Titanium, Quartz, Polyurethane, Delrin, Silicone, Grease, RTV, Stainless Steel

12.B.8 Transducer Installation. When a transducer is installed in a well with a pump, it is important to install an access tube with the pump column. This will prevent the transducer cable from becoming entangled with the pump column. A tubing ID of 1-inch minimum is recommended.

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V QA Objectives and Criteria

Item #2 - Revised Table 4-1 to include soil gas chromatograph specifications.

Item #3a - Refer to attached methods for soil gas and OP21-16-8010.

Item #3b - Added Tables 4-2a GC Detection Limits,
4-2b GC Soil Gas Detection Limits

VIII Calibration Procedures and Frequency

Item #4 - Refer to attached methods for soil gas.

IX Analytical Procedures

Items #1, #6, refer to attached methods for soil gas
OP 21-16-8010.

X Data Reduction, Validation and Reporting

Items #4, #5 reporting

The TCL/TAL soil and water samples will include a CLP equivalent deliverable package.

The deliverable package for samples analyzed by the 601 method will include:

- a. Forms I (VOCs)
- b. All sample and standard chromatograms and quantitation lists.
- c. All calculations associated with sample results.
- d. Chromatograms and quantitation lists for method blank and duplicate analyses.
- e. Response Factor calculations (initial and continuing).
- f. Instrument run logs.
- g. A narrative summary including instruments employed and operating conditions (temperature program, integrator settings, and electronic parameters).

XI Internal QC Checks

Items #4, #5 - Refer to attached methods for soil gas
OP-21-16-8010.

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XII Performance and System Audits, Items #4, #6

The QA Manager will conduct two field audits during RI activities, to confirm that SOPs are being observed. Any deficiencies will be documented and corrective action will be approved and initiated by the Site Manager. Deviations from the Health and Safety Plan will be discussed with the Site Health and Safety Officer and the Site Manager and will be corrected as soon as possible so that work can continue. The first audit will be scheduled during the start-up of activities, to ensure that field personnel are familiar with the SOPs, and the second scheduled during the final stage of activities.

Performance audits to assess the accuracy of the field gas chromatograph will be conducted at the discretion of the QA Manager.

With regard to laboratory audits, samples for the Commodore project will be collected during the last quarter of 1989. Areas of the laboratory scheduled for systems audits during this quarter include GC/MS and sample log in. Since the requirements for this project are not beyond normal laboratory criteria for CLP-type programs, additional client-specific requirements will not be necessary. An updated version of Table 4-3 is included.

Additional client-specific performance samples are not anticipated for this project.

XIV Specific SOPs Used to Assess Data Precision, Accuracy, Representativeness and Completeness

The data quality objectives for the field activities of the program are to provide accurate and representative subsurface information on the Commodore site and the vicinity, with emphasis on groundwater data. Nine groundwater samples will be collected from the newly installed monitoring wells in addition to the 16 previously installed monitoring wells, irrigation wells and public supply wells. The new well samples are considered critical and will be assessed on an individual basis as they will be collected from the only wells in the area that are completed in discreet zones. Therefore, if QA requirements are not met, the samples will be reanalyzed. If sample QA criteria are still not acceptable (e.g. surrogate recoveries are outside limits or samples are lost or broken) the well will be resampled.

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For completeness considerations, 100 percent of the samples collected from new monitor wells must meet QA requirements and sampling SOPs.

The remainder of the data will be assessed by matrix type. If 80 percent of sample QA requirements are met, the matrix sample results will be considered acceptable. If QA criteria are met for less than 80 percent of the sample group, the data will be qualified when used in contouring and modeling. These actions will also apply where samples are lost or holding times are exceeded. In any case, all deviations from QA requirements will be documented and corrective action approved by the Site Manager. For a group of samples other than groundwater samples collected from completed monitor wells, an 80 percent completeness record will be considered acceptable.

The groundwater data will be used to model concentrations of compounds; the soil gas and soil boring information will be evaluated with respect to determining the optimal locations of the monitoring wells and contoured to determine the location and extent of the compounds in the soil. As the end uses of the data are not related to regulatory action levels, (i.e. a certain level requires that an additional data point be sampled) any deviations from the 30 percent goal of precision and accuracy encountered in QA criteria may be addressed as a question of completeness of the data set.

XV Corrective Action

Problems associated with data collection may include unacceptable calibration of the field gas chromatograph unit and decontamination of the soil gas system. These problems are quantifiable and are addressed in SOPs relating to the gas chromatograph and the soil gas unit. Any deviations will be documented by the technical support staff operating the GC, who will initiate corrective action with the acknowledgement of the Site Manager.

Accurate calibration is essential to the results of the borehole geophysical logging. The manufacturer's instructions will be followed for calibration; deviations will be documented by the operator and incorporated into the final data analysis.

The instruments used during the packer testing are designed to provide information on equilibration of the system. If the system does not stabilize, indicating a problem with the packers or a poor seal with the borehole

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wall, the situation will be documented and the packer assembly will be repositioned or withdrawn for inspection, with approval of the Site Manager.

The remaining RI activities planned at the Commodore site do not include quantifiable measures of defects in the operations. In addition, it may not be possible to determine whether the cause of a problem, such as less than 100 percent core or drill cuttings recovery, is due to subsurface conditions or operator influence. During such activities, deviations from optimal conditions will be documented in the field log book by the technical support staff. It will be the decision of the support staff as to whether corrective action, such as changing penetration rates, is required, which will be approved and initiated by the Site Manager.

XVI QA Reporting Procedures

Any changes in the QAPP will be discussed with the Project Manager, initiated by the Site Manager and approved by EPA. A distribution list will be added after the RISOP Table of Contents listing recipients of revised versions.

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STANDARD OPERATING PROCEDURES OF THE ON-SITE LABORATORY FOR
THE ANALYSIS OF SOIL GAS

o Analysis for Selected Volatile Contaminants

All samples received by the on-site laboratory will be analyzed by gas chromatography using an Electron Capture Detector for 1,2-Dichloroethane, Trans-1,2-Dichloroethene, 1,1,1-Trichloroethane, Trichloroethene, and Tetrachloroethene. Appropriate instrument conditions will be used to obtain best sensitivity and separation of target compounds.

Injection of samples into the gas chromatograph will be accomplished by gas sample loops or by the manual injections using air-tight syringes.

o Calibration

Liquid standards prepared from neat will be used to generate a 3-point calibration curve weekly, for each GC detector. Percent relative standard deviation (RSD) is not to exceed 30 percent. Midpoint check standards will be run periodically throughout the day (minimum every 8 hours). Should a check standard fall out of calibration by more than ± 20 percent, a new 3 point standard curve will be run. A check standard may be repeated if it falls outside the ± 20 percent window of the curve to assure that the sensitivity of the instrument has in fact changed.

o QA/QC Samples

During the course of the project the following QA/QC measures will be adhered to:

- o Laboratory QA/QC
 - 10 percent of the samples will be analyzed in replicate.
 - 5 percent of the deconned bulbs will be analyzed.
- o Field QA/QC
 - Samples will be taken with 5 percent duplication.

Replicates will be the same sample analyzed twice by the ECD Gas Chromatograph. Replicates are used to show the precision of the analytical method and will be reported as a relative percent difference. Duplicates will be two

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individual samples taken at the same grid location. Duplicates are used to show the precision of the sampling method and site conditions and will be reported as a relative percent difference. Decon bulb checks will be the analysis of bulbs that have been through the Decon process to assure no carry over from one sample to the next.

QA/QC objectives are summarized in Table 4-2b.

o Detection Limits

The detection limit for trichloroethane, trichloroethene, and tetrachloroethene is 0.5 ng of analyte per 1 ml analyzed on column. This can be represented as a theoretical detection limit of 0.1 ppm v/v. Trans-1,2-dichloroethene and 1,2-dichloroethane will have a detection limit of 5 ng on column or 1 ppm v/v.

o Dilutions

Sample dilutions will be carried out by adjusting the volume injected or by making variable dilutions in a 125 ml gas sampling bulb.

o Reporting

All data will be tabulated on spreadsheets. Calibration criteria and decon bulbs will also be reported separately from the field samples so that QA/QC criteria may be closely monitored.

o Decon Procedures

- Bulb decon

Bulbs will be deconned in one of two ways. In the case of minimum contamination bulbs will be attached to a sampling pump and purged with room air for ten minutes. A 1200 watt hairdryer will be used to heat the bulbs up during purging. In cases where high levels of contamination are present the bulbs can be deconned by heating in an oven set at 100 degrees C. The heated bulbs will be purged under vacuum with the inlet air going through an activated charcoal filter.

- Probe decon

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Copper tubing will be washed with water and wiped dry with paper towels. Teflon tubing will be removed, wiped clean and heated overnight at 100 degrees C.

Any unforeseen problem or site specific adjustments that need to be addressed will be handled by the on-site lab manager and field crews. The corrective actions taken will be signed off on by key personnel.¹ Notes of any deviation of this SOP will be entered into the lab notebook, dated, and signed. All notebooks will be dated and initialed by the appropriate technician and reviewed daily by the lab manager who will also date and initial. Changes to the protocols will be discussed with the project manager and documented before changes are made.

1 (Site Manager, Lab Manager, and verbal from Project Manager).

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Table 4-3
External Performance and Systems Audits
WESTON Analytical Laboratories

Agency	Parameters	Type	Frequency	Required For
Illinois EPA	WS-WP	Performance System	Semi-annually Every 2 Yrs.	Water/Waste Water Cert.
NY Dept of Health	WS-WP	Performance	Semi-annually Annual	Water/Waste Water Cert.
NY State Dept. Env. Conserv.	Inorganic- Organic TCL	Performance System	EPA CLP Qtrly Blind	State Analytical Contract
NJ Dept. of Environ. Protection	WS-WP	Performance System	Annually Every 2 Yrs.	Water/Waste Water Cert.
	Haz. Waste	Performance	EPA CLP Qtrly	Haz. Waste Approval
Oklahoma WRB	WS-WP	Performance	Semi-annually	Water/Waste Water Cert.
PA Dept. of Environ. Res.	WS	Performance System	Annually Every 2 Yrs.	Water Cert.
	Haz. Waste	Performance	EPA CLP Qtrly	State Anal. Contract
U.S. EPA	Inorganic- Organic TCL	Performance System	Quarterly Every 2 Yrs.	Superfund Related Analytical Work
U.S. Army Corps of Engineers (DERA)	Inorganic- Organic	Performance System	As Contract Requires	Water/Waste Water, Superfund Analytical Work

Last on-site by U.S. EPA was performed in May 1988.

Last PA DER on-site was performed in May 1987, and last IEPA on-site was performed in September 1988.

WS = Water Supply (Drinking Water)

WP = Water Pollution (Wastewater)

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Table

Summary of Precision, Accuracy, and Completeness Objectives

Sample Matrix	Analysis	Analytical Method	Reference	% RPD Precision	% R Accuracy	Completeness
Soil	GC Volatiles ¹	EPA 8010	EPA SW846 ²	30%	30%	80 percent
Water	GC Volatiles ¹	EPA 601	40 CFR 136 ³	30%	30%	80 percent
Soil Gas	GC Volatiles ¹			30%	Continuing calibration 20%	80 - 100 percent
Soil	TCL Volatiles	EPA CLP	EPA CLP SOW (organics) ⁴	30%	(6)	80 percent
Water	TCL Volatiles	EPA CLP	EPA CLP SOW (organics) ⁴	(6)	(6)	80 percent
Soil	TCL Semivolatiles	EPA CLP	EPA CLP SOW (organics) ⁴	(6)	(6)	80 percent
Water	TCL Semivolatiles	EPA CLP	EPA CLP SOW (organics) ⁴	(6)	(6)	80 percent
Soil	TCL Pesticides/PCB	EPA CLP	EPA CLP SOW (organics) ⁴	(6)	(6)	80 percent
Water	TCL Pesticides/PCB	EPA CLP	EPA CLP SOW (organics) ⁴	(6)	(6)	80 percent
Soil	TAL Metals	EPA CLP	EPA CLP SOW (inorganics) ⁵	(6)	(6)	80 percent
Water	TAL Metals	EPA CLP	EPA CLP SOW (inorganics) ⁵	(6)	(6)	80 percent
Soil	Cyanide	EPA CLP	EPA CLP SOW (inorganics) ⁵	(6)	(6)	80 percent
Water	Cyanide	EPA CLP	EPA CLP SOW (inorganics) ⁵	(6)	(6)	80 percent

¹See Table 4-2a, 4-2b for complete list of target compounds.

²Test Methods for Evaluating Solid Waste U.S. EPA, SW-846, Nov. 1986, 3rd Edition.

³U.S. EPA "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, Final Rule and Interim Final Rule and Proposed Rule," 40 CFR 136, 10/26/84 Federal Register.

⁴U.S. EPA Contract Laboratory Program "Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration," 2/88 and as revised through 5/89.

⁵U.S. EPA Contract Laboratory Program "Statement of Work for Inorganic Analysis, Multi-Media, Multi-Concentration," 7/88 and as revised through 2/89.

⁶Precision and accuracy are set on a compound-specific basis.

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Table 4-2a

GC Volatile Parameters and Detection Limits

	Matrix: Units:	Water ug/L	Soil ug/kg
Chloromethane		2	2
Bromomethane.....		2	2
Vinyl Chloride.....		2	2
Chloroethane.....		2	2
Methylene Chloride.....		4	4
Trichlorofluoromethane.....		1	1
1,1-Dichloroethene.....		1	1
1,1-Dichloroethane.....		1	1
*Trans-1,2-Dichloroethene.....		1	1
Chloroform.....		1	1
*1,2-Dichloroethane.....		1	1
*1,1,1-Trichloroethane.....		1	1
Carbon Tetrachloride.....		1	1
*Bromodichloromethane.....		1	1
1,2-Dichloropropane.....		1	1
*Trans-1,3-Dichloropropene.....		1	1
Trichloroethene.....		1	1
Dibromochloromethane.....		1	1
1,1,2-Trichloroethane.....		1	1
*Benzene.....		1	1
*cis-1,3-Dichloropropene.....		1	1
2-Chloroethylvinylether.....		1	1
*Bromoform.....		1	1
Tetrachloroethene.....		1	1
*1,1,2,2-Tetrachloroethane.....		1	1
*Toluene.....		1	1
Chlorobenzene.....		1	1
*Ethylbenzene.....		1	1
1,2-Dichlorobenzene.....		1	1
1,3-Dichlorobenzene.....		1	1
1,4-Dichlorobenzene.....		1	1

*Matrix spike compound.

**Units = ug/L for wter, ug/kg for soil.

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Table 4-2b

QA/QC Objectives For Soil Gas Survey
 Organic Contaminants

Parameter To Measure	TCA, TCE, PCE	1,2-DCA <u>Trans-1,2-DCE</u>
Units	PPM V/V	PPM V/V
MDL	0.1 ppm	1 ppm
Calibration* Criteria RSD	30%	30%
Continuing* Calibration %	20%	20%
Precision** RPD (Replicates)	30%	30%
Precision** RPD (Duplicates)	30%	30%

*Exceeding these criteria will warrant re-calibration.

**These goals will demonstrate data useability and quality without qualification. Data outside these objectives will be qualified and evaluated on a case to case basis with respect to the project goals.

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ORGANIC ANALYSIS PROTOCOLS
PURGEABLE HALOCARBONS & PURGEABLE AROMATICS
BY PURGE AND TRAP GC

1.0 SCOPE AND APPLICATION

- 1.1 This method covers the determination of the 29 purgeable halocarbons and 7 purgeable aromatics listed in Appendix A. Additional compounds can be added after the verification of detection limits and retention times. WESTON OP21-16-8010 and OP21-16-8020 are identical.
- 1.2 The method detection limit for each parameter is listed in Appendix A.

2.0 SUMMARY OF METHOD

- 2.1 An inert gas is bubbled through a 5 mL water sample contained in a specially designed purging chamber at ambient temperature. The analytes are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the analytes are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb analytes onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the analytes which are then detected with an appropriate detector.

3.0 SAFETY

- 3.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material, as well as, additional references to laboratory safety, are available from the Health and Safety Officer.
- 3.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens, carbon tetrachloride, chloroform, 1,4,-dichlorobenzene, vinyl chloride, and benzene. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

4.0 APPARATUS AND MATERIALS

- 4.1 Purge and trap system - The purge and trap system consists of three separate pieces of equipment: a purging device, trap, and

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desorber. Several complete systems are now commercially available.

- 4.1.1 The purging device must be designed to accept 5 mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Larger purging devices capable of handling increased sample volumes may be substituted to enhance instrument detection limits.
- 4.1.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. The trap must be packed to contain the following minimum lengths of adsorbents 1.0 cm of methyl silicone coated packing, 7.7 cm of 2,6-diphenylene oxide polymer 7.7 cm of silica gel, 7.7 cm of coconut charcoal.
- 4.1.3 The desorber must be capable of rapidly heating the trap to 180°C. The polymer section of the trap should not be heated higher than 180°C and the remaining sections should not exceed 200°C.
- 4.1.4 The purge and trap system may be assembled as a separate unit or be coupled to a gas chromatograph.
- 4.2 Gas Chromatograph - An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.
 - 4.2.1 Column 1 - 8 ft. long x 0.1 in. ID stainless steel or glass, packed with 1% SP-1000 on Carbopack B (60/80 mesh) or equivalent.
 - 4.2.2 Column 2 - 6 ft. long x 0.1 in. ID stainless steel or glass, packed with chemically bonded n-octane on Porasil-C (100/120 mesh) or equivalent.
 - 4.2.3 Alternate columns may be used after the verification of retention times and performances.
- 4.3 Detectors - electrolytic conductivity detector (for halocarbons) and photoionization detector (for aromatics). These detectors have proven effective in the analysis of waste waters for the parameters listed in Appendix A. Alternate detectors may be used after the verification of detection limits and performance criteria.
- 4.4 Syringes - 5 mL glass hypodermic with Luerlok tip (two each), if applicable to the purging device.

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- 4.5 Micro syringes - 1 uL, 10 uL, 20 uL, 50 uL and 100 uL.
- 4.6 Syringe valve - 2-way, with Luer ends (three each).
- 4.7 Syringe - 5 mL, gas tight.
- 4.8 Bottle - 15 mL, screw-cap, with Teflon cap liner.
- 4.9 Balance - Analytical, capable of accurately weighing 0.0001 g.
- 5.0 REAGENTS
- 5.1 Reagent water - Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.
 - 5.1.1 Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 lb of activated carbon (Filtrisorb-300, Calgon Corp., or equivalent). If traces of methylene chloride are present in reagent water, it is heated at 75°C for about an hour to get rid of this contamination.
 - 5.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.
 - 5.1.3 Reagent water may also be prepared by boiling water for 15 min. Subsequently, while maintaining the temperature at 90°C, bubble a contaminant-free inert gas through the water for 1 hour. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum cap.

Note: Option 5.1.3 is not permitted for NJDEP analysis.
- 5.2 Trap Materials
 - 5.2.1 Coconut charcoal-6/10 mesh sieved to 26 mesh, Barnebey Cheney, CA-580-26 lot #M-2649 or equivalent.
 - 5.2.2 2,6-Diphenylene oxide polymer- Tenax, (60/80 mesh), chromatographic grade or equivalent.
 - 5.2.3 Methyl silicone packing-3% OV-1 on Chromosorb-W (60/80 mesh) or equivalent.
 - 5.2.4 Silica gel-35/60 mesh, Davison, grade-15 or equivalent.
- 5.3 Methanol-Pesticide quality or equivalent.
- 5.4 Stock standard solutions - Stock standard solutions may be prepared from pure standard material or purchased as certified solutions. Prepare stock standard solutions in methanol using assayed liquids or gases as appropriate. Because of the toxicity

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of some of the compounds, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used

- 5.4.1 Place about 9.8 mL of methanol into a 10 mL ground glass stoppered volumetric flask. Allow the flask to stand unstoppered, for about 10 min. or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.
- 5.4.2 Add the assayed reference material:
 - 5.4.2.1 Liquid - Using a 100 uL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. Be sure that the drops fall directly into the alcohol without contacting the neck of the flask.
 - 5.4.2.2 Gases - To prepare standards for any of the six halocarbons that boil below 30°C (bromomethane, chloroethane, chloromethane, dichlorodifluoromethane, trichlorofluoromethane, vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0-mL mark. Lower the needle to 5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid (the heavy gas will rapidly dissolve into the methanol).
- 5.4.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in ug/uL from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.
- 5.4.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at -10 to -20°C and protect from light.
- 5.4.5 Prepare fresh standards weekly for the six gases. All other standards must be replaced after one month, or sooner if comparison with check standards indicates a problem.
- 5.4.6 Secondary dilution standards - using stock standard solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.0 CALIBRATION

6.1 Assemble a purge and trap system that meets specifications in Section 4.1. Condition the trap overnight at 180°C by backflushing with an inert gas flow of at least 20 mL/min. Condition the trap for 10 min. once daily prior to use.

6.2 Connect the purge and trap system to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate conditions equivalent to those given in Table 1. For columns other than those listed in Table 1, conditions must be adjusted for optimum separation of the compounds of interest. Calibrate the purge and trap gas chromatographic system using the external standard technique.

6.3 External Standard Calibration Procedure

6.3.1 Prepare calibration standards at a minimum of 5 concentration levels for each parameter by adding the appropriate volume of one or more secondary dilution standards to reagent water. The standard can be added to the water in a volumetric flask or the water contained in the syringes typically used for the transfer of samples. One of the external standards must be at concentration at or below the detection limit (Appendix A) and the other concentrations should correspond to the working range of the detector. These standards must be used immediately after preparation.

6.3.2 Analyze each calibration standard according to Section 9, and tabulate peak height or area responses versus the concentration in the standard. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve. Calibration data must be approved by a supervisor for the correct range of calibration standards and verification of instrument performance.

6.3.3 The accuracy of the calibration must be determined by the analysis of an EMSL check sample containing at least 25% of all of the parameters for which the instrument is being calibrated. The results for each parameter must fall within the acceptance limits supplied with the check sample or within $\pm 15\%$ of the true value if acceptance limits are not supplied. A standard prepared from a different stock than the calibration standards can be substituted for the EMSL check sample, with acceptance limits $\pm 15\%$. The compounds verified during this calibration check must be varied so that all compounds in Appendix A are evaluated intermittently.

- 6.4 The working calibration curve, calibration factor or RF must be verified at the beginning of each shift by the analysis of a 20 ug/L check standard. The response must also be verified during the shift by the analysis of a 20 ug/L check standard at a frequency of at least once every 10 samples.
- 6.4.1 Analyze the check standard according to Section 9, *QC*.
- 6.4.2 For each parameter, compare the response (Q) with the corresponding calibration acceptance criteria found in Table 2. If the responses for all parameters of interest fall within the designated ranges analysis of actual samples can continue. If any individual (Q) falls outside the range, proceed according to Section 6.4.3.
- Note: The calibration acceptance criteria for compounds not listed in Table 2 will be $\pm 25\%$ of the true value. Different ranges for calibration acceptance criteria can be substituted for the values in Table 2 to comply with a project-specific QAPP.
- 6.4.3 Repeat the test only for those parameters that failed to meet the calibration acceptance criteria. If the response for a parameter does not fall within the range in this second test, a new calibration curve, calibration factor, or RF must be prepared for that parameter.

7.0 QC PROGRAM

- 7.1 The analyst must monitor both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample with a surrogate standard containing bromochloromethane (purgeable halocarbon surrogate) and trifluorotoluene (purgeable aromatic surrogate). The surrogate standard will be added to the sample to provide a final concentration of 20 ug/L for a particular sample/blank aliquot being purged. The surrogate result will be reported on a percent recovery basis, and it will be determined from a 3-level calibration curve of 10, 20 and 40 ug/L of the surrogate standard, generated at the time of calibration of the other parameters.

Any sample or blank having a surrogate recovery outside of the laboratory control limits (Table 3) must be re-analyzed. If the surrogate recovery for the re-analysis of a blank is outside the laboratory control limits, the unit leader or an analyst appointed by the unit leader must be notified. If the surrogate recovery for the re-analysis of a sample is outside laboratory control limits but the instrument is in control (as demonstrated through the analysis of the check standard and the surrogate recoveries for the blank) then the out of control surrogate recoveries for the sample will be attributed to a matrix effect.

- 7.2 Method performance will also be monitored through the analysis of matrix spike and matrix spike duplicate samples. These samples will be analyzed at a frequency of at least 10% for client sample batches of 10 or more samples or at least one time per week. The compounds to be spiked into the sample are designated in the Table in Appendix A. The spike should be at a level of 20 ug/L or 1 to 5 times higher than the background concentration of the parameters in the sample.
- 7.2.1 Analyze one 5-mL sample aliquot to determine the background concentration (B) of each parameter. Spike two separate 5-mL sample aliquots with the appropriate volume of the matrix spike concentrate and then analyzed the spiked samples to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as $100 (A-B)\%/T$, where T is the known true value of the spike.
- 7.2.2 As part of the QC program for the laboratory, method accuracy and precision for sample matrices must be assessed. Results for all matrix spike samples will be reported to the QC department for the generation of control charts.
- 8.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING
- 8.1 All samples must be iced or refrigerated from the time collection until analysis.
- 8.2 Grab samples must be collected in glass containers having a total volume of at least 25 mL. Fill the sample bottle just to overflowing in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottles so that no air bubbles are entrapped in it. If preservative has been added, shake vigorously for 1 min. Maintain the hermetic seal on the sample bottle until time of analysis.
- 8.3 All samples must be analyzed for purgeable halocarbons within 14 days of collection. All samples must be analyzed for purgeable aromatics within 7 days of collection. If the purgeable aromatics sample has been preserved at the time of collection by the addition of 1:1 HCl (until a sample pH of about 2 has been achieved), then the holding time for purgeable aromatics can be extended to 14 days from the time of collection.
- 9.0 PROCEDURE
- 9.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are estimated retention times that can be achieved under these conditions. Other packed columns, chromatographic conditions, or detectors may be used if the detection limit requirements are met.
- 9.2 Calibrate the system daily as described in Section 5.

- 9.3 Adjust the purge gas (nitrogen or helium) flow rate to 40 mL/min. Attach the trap inlet to the purging device, and set the purge and trap system to purge. Open the syringe valve located on the purging device sample introduction needle.
- 9.4 Allow the sample to come to ambient temperature prior to introducing it to the syringe. Remove the plunger from a 5 mL syringe and attach a closed syringe valve. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of the overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. This process destroys the validity of the sample for future analysis. Add the appropriate volume of the surrogate spiking standard to the sample contained in the syringe.
- 9.5 Immediately attach the syringe to the syringe valve on the purging device and inject the sample into the purging chamber.
- 9.6 Close both valves and purge the sample for 11.0 ± 0.1 min. at ambient temperature.
- 9.7 After the 11-min purge time, attach the trap to the chromatograph, adjust the purge and trap system to the desorb mode, and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180°C while backflushing the trap with an inert gas between 20 and 60 mL/min. for 4 min. If rapid heating of the trap cannot be achieved, the GC column must be used as a secondary trap by cooling it to 30°C (subambient temperature, if poor peak geometry or random retention time problems persist) instead of the initial program temperature of 45°C.
- 9.8 After desorbing the sample for 4 min, recondition the trap by returning the purge and trap system to the purge mode. Wait 15 s then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180°C. After approximately 7 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When the trap is cool, the next sample can be analyzed.
- 9.9 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

If the entire list of purgeable halocarbons and purgeable aromatic parameters are to be determined, there is the possibility that several of the parameters will co-elute with a given chromatographic column, making it impossible for the analyst to identify one parameter of the other by retention time. If this situation occurs during sample analysis, the analyst must have the sample re-analyzed with a different chromatographic column that provides adequate separation of the parameters in question, or have the samples analyzed by GC/MS.

9.10 If the response for a peak exceeds the working range of the system, prepare a dilution of the sample with reagent water from the aliquot in the second syringe and reanalyze.

9.11 Confirmation of parameters positively identified with the initial analysis will be performed by analysis on a second, dissimilar chromatographic column, or by GC/MS, only at the request of the client.

10.0 CALCULATIONS

10.1 Determine the concentration of individual compounds in the sample. Calculate the concentration of the parameter being measured from the peak response using the calibration curve calibration factor determined in Section 6.

11.0 MODIFICATION FOR SOIL AND WASTE SAMPLES

11.1 Water-miscible liquids:

11.1.1 Water-miscible liquids are analyzed as water samples after first diluting them at least 50-fold with reagent water.

11.1.2 Initial and serial dilutions can be prepared by pipetting 2 mL of the sample to a 100-mL volumetric flask and diluting to volume with reagent water. Transfer immediately to a 5-mL gas-tight syringe.

11.1.3 Alternatively, prepare dilutions directly in a 5-mL syringe filled with reagent water by adding at least 20 uL, but not more than 100 uL of liquid sample. The sample is ready for addition of surrogate and, if applicable, internal and matrix spiking standards.

11.2 Sediment/soil and waste samples: It is highly recommended that all samples of this type be screened prior to the purge and trap GC analysis. These samples may contain percent quantities of purgeable organics that will contaminate the purge and trap system, and require extensive cleanup and inspection downtime. Use the screening data to determine whether to use the low-level method (0.005-1 mg/kg) or the high-level method (>1 mg/kg).

- 11.2.1 Low-level method: This is designed for samples containing individual purgeable compounds of <1 mg/kg. It is limited to sediment/soil samples and waste that is of a similar consistency (granular and porous). The low-level method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogate and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples.
- 11.2.1.1 Use a 5-g sample if the expected concentration is <0.1 mg/kg or a 1-g sample for expected concentrations between 0.1 and 1 mg/kg.
- 11.2.1.2 The GC system should be set up as in Section 9.0. This should be done prior to the preparation of the sample to avoid loss of volatiles from standards and samples.
- 11.2.1.3 Remove the plunger from a 5-mL Luerlock type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5.0 mL. Add the appropriate volume of the surrogate spike to the syringe. Matrix spiking solutions, if indicated, should be added to the sample at this time.
- 11.2.1.4 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh the amount determined in Paragraph 11.2.1.1 into a tared purge device. Note and record the actual weight to the nearest 0.1 g.
- 11.2.1.5 Sample results are reported based on a dry-weight basis. A portion of sample for moisture determination should be weighed out at the same time as the portion used for analytical determination. Immediately after weighing the sample for extraction, weigh 5-10 g of the sample into a tared crucible. Determine the percent moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing:

$$\frac{\text{g of sample} - \text{g of dry sample}}{\text{g of sample}} \times 100 = \% \text{moisture}$$

- 11.2.1.6 Add the spiked reagent water to the purge device, which contains the weighed amount of sample, and connect the device to the purge and trap system.

Note: Prior to the attachment of the purge device, steps 11.2.1.5 and 11.2.1.6 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

- 11.2.1.7 Proceed with the analysis as outlined in Section 9. Use 5 mL of the same reagent water as in the reagent blank. If saturated peaks occurred or would occur if a 1-g sample were analyzed, the high-level method must be followed.
- 11.2.2 High-level method: This method is based on extracting the sediment/soil with methanol. A waste sample is either extracted or diluted, depending on its solubility in methanol. An aliquot of the extract is added to reagent water containing surrogate and, if applicable, matrix spiking standards. All samples with an expected concentration of >1.0 mg/kg should be analyzed by this method.
- 11.2.2.1 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. For sediment/soil and waste that are insoluble in methanol, weigh 5 g (wet weight) of sample into a tared 20-mL vial. Use a top-loading balance. Note and record the actual weight to 0.1 gram and determine the percent moisture of the sample using the procedure in Paragraph 11.2.1.5. For waste that is soluble in methanol, weigh 1 g (wet weight) into a tared scintillation vial or culture tube or a 10-mL volumetric flask. (If a vial or tube is used, it must be calibrated prior to use.) Pipet 10.0 mL of methanol into the vial and mark the bottom of the meniscus. Discard this solvent.)
- 11.2.2.2 Quickly add 9.0 mL of methanol; then add 1.0 mL of the surrogate spiking solution to the vial. Cap and shake for 2 minutes.
- Note: Steps 11.2.2.1 and 11.2.2.2 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.
- 11.2.2.3 Pipet approximately 1 mL of the extract to a GC vial for storage, using a disposable pipet. The remainder may be disposed of. Transfer approximately 1 mL of reagent methanol to a separate GC vial for use as the method blank for each set of samples. These extracts may be sorted at 4°C in the dark, prior to analysis.
- 11.2.2.4 The GC system should be set up as in Section 9. This should be done prior to the addition of the methanol extract to reagent water.
- 11.2.2.5 Table 4 can be used to determine the volume of methanol extract to add to the 5 mL of reagent water for analysis. If a screening procedure was followed, use the estimated concentration to determine the appropriate volume. Otherwise, estimate the concentration range of the sample from the low-level analysis to determine the appropriate volume. If the sample was submitted as a high-level sample, start with 100 uL. All dilutions must keep

the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

- 11.2.2.6 Remove the plunger from a 5.0 mL Luerlock type syringe equipped with syringe valve and fill until overflowing with a reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 mL. Pull the plunger back to 5.0 mL to allow volume for the addition of the sample extract and of standards. Add 10 uL of internal standard solution. Also add the volume of methanol extract determined in Paragraph 11.2.2.5 and a volume of methanol solvent to total 100 uL (excluding methanol in standards).
- 11.2.2.7 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valve and inject the water/methanol sample into the purging chamber.
- 11.2.2.8 Proceed with the analysis as outlined in Section 9. Analyze all reagent blanks on the same instrument as that used for the sample. The standards and blanks should also contain 100 uL of methanol to simulate the sample conditions.
- 11.2.2.9 For a matrix spike in the high-level sediment/soil samples, add 8.0 mL of methanol, 1.0 mL of surrogate spike solution and 1.0 mL of matrix spike solution. Add a 100 uL aliquot of this extract to 5 mL of water for purging (as per Paragraph 11.2.2.6).

12.0 INSTRUMENT PERFORMANCE, MAINTENANCE AND DOCUMENTATION

- 12.1 Acceptable instrument performance will be verified during the calibration procedure. The linear response of the detector is verified by the analysis of at least 5 calibration standards. The low level standard must be at or below the detection limits listed in Table 1. This low level standard demonstrates that the instrument and detector are capable of meeting the detection limit requirement. The high concentration standard defines the upper working range for the instrument for that particular calibration. The chromatographic column will also be evaluated with respect to the chromatograms obtained from the calibration. These evaluations will be made relative to the performance predicted by the manufacturer and historical data collected by the laboratory for similar chromatographic columns.
- 12.2 Routine maintenance of the instrumentation will include cleaning of the purge and trap glassware, cleaning of the detector and replacement of the Teflon ferrules on the purge and trap device. The glassware and syringes used for the handling of the samples will be cleaned by a deionized water rinse followed by an oven bake at 145°C for at least 30 minutes. Each clean syringe and purge chamber will be used for the processing of one sample. The requirement for detector cleaning and/or ferrule replacement is

indicated by a decrease in the expected response for surrogate and/or calibration check compounds. Detector cleaning is performed as per the manufacturer's recommended procedure. The frequency of these maintenance tasks will be dictated by the number of samples analyzed and the condition of the samples. Replacement of the trap and GC column will be required less frequently, and will be indicated by a deterioration in the overall performance of the instrument. These tasks will be performed at the discretion of the unit leader and/or an analyst designated by the unit leader.

- 12.3 Log books will be maintained for each instrument containing the appropriate standard/sample identification information, the date(s) and time(s) of analysis, information on sample dilutions, and a verification of the client's sample identification. Maintenance information will also be documented in the comments section of the logbook, as it is performed.

APPENDIX A - GC Volatile Parameters and Detection Limits

	Matrix:	Water	Soil	MDL
	Units:	ug/L	ug/kg	**
Chloromethane	2	2	0.7	
Bromomethane	2	2	6	
Vinyl Chloride	2	2	0.2	
Chloroethane	2	2	3	
Methylene Chloride	4	4	0.1	
Trichlorofluoromethane	1	1	0.1	
1,1-Dichloroethene	1	1	0.1	
1,1-Dichloroethane	1	1	0.1	
*Trans-1,2-Dichloroethene	1	1	0.2	
Chloroform	1	1	0.1	
*1,2-Dichloroethane	1	1	0.2	
*1,1,1-Trichloroethane	1	1	0.2	
Carbon Tetrachloride	1	1	0.1	
*Bromodichloromethane	1	1	0.2	
1,2-Dichloropropane	1	1	0.2	
*Trans-1,3-Dichloropropene	1	1	0.1	
Trichloroethene	1	1	0.1	
Dibromochloromethane	1	1	0.1	
1,1,2-Trichloroethane	1	1	0.1	
*Benzene	1	1	0.1	
*cis-1,3-Dichloropropene	1	1	0.2	
2-Chloroethylvinylether	1	1	0.5	
*Bromoform	1	1	0.5	
Tetrachloroethene	1	1	0.2	
*1,1,2,2-Tetrachloroethane	1	1	0.3	
*Toluene	1	1	0.1	
Chlorobenzene	1	1	0.1	
*Ethylbenzene	1	1	0.1	
1,2-Dichlorobenzene	1	1	0.5	
1,3-Dichlorobenzene	1	1	0.5	
1,4-Dichlorobenzene	1	1	0.1	

*Matrix spike compound

**Units=ug/L for water, ug/kg for soil

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TABLE 1 - CHROMATOGRAPHIC CONDITIONS

Column 1 Conditions:	Carbopack B (60/80 mesh) coated with 1% SP-1000 packed in an 8 ft. x 0.1 in. ID stainless steel or glass column with helium carrier gas at 40 mL/min. flowrate. Column temperature held at 45°C for 3 min. then programmed at 8°C/min. to 220°C and held for 15 min.
Column 2 Conditions:	Porisil - C (100/120 mesh) coated with n-octane packed in a 6 ft. x 0.1 in. ID stainless steel or glass column with helium carrier gas at 40 mL/min. flowrate. Column temperature held at 50°C for 2 min. then programmed at 6°C/min. to 170°C and held for 4 min.

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TABLE 2 - Calibration and QC Acceptance Criteria

Purgeable Aromatics

Parameter	Range for Q (ug/L)
Benzene	15.4-24.6
Chlorobenzene	16.1-23.9
1,2-Dichlorobenzene	13.6-26.4
1,3-Dichlorobenzene	14.5-25.5
1,4-Dichlorobenzene	13.9-26.1
Ethylbenzene	12.6-27.4
Toluene	15.5-24.5

Purgeable Halocarbons

Parameter	Range for Q (ug/L)
Bromodichloromethane	15.2-24.8
Bromoform	14.7-25.3
Bromomethane	11.7-28.3
Carbon tetrachloride	13.7-26.3
Chlorobenzene	14.4-25.6
Chloroethane	15.4-24.6
2-Chloroethylvinyl ether	12.0-28.0
Chloroform	15.0-25.0
Chloromethane	11.9-28.1
Dibromochloromethane	13.1-26.9
1,2-Dichlorobenzene	14.0-26.0
1,3-Dichlorobenzene	9.9-30.1
1,4-Dichlorobenzene	13.9-26.1
1,1-Dichloroethane	16.8-23.2
1,2-Dichloroethane	14.3-25.7
1,1-Dichloroethene	12.6-27.4
trans-1,2-Dichloroethene	12.8-27.2
1,2-Dichloropropane	14.8-25.2
cis-1,3-Dichloropropene	12.8-27.2
trans-1,3-Dichloropropene	12.8-27.2
Methylene chloride	15.5-24.5
1,1,2,2-Tetrachloroethane	9.8-30.2
Tetrachloroethene	14.0-26.0
1,1,1-Trichloroethane	14.2-25.8
1,1,2-Trichloroethane	15.7-24.3
Trichloroethene	15.4-24.6
Trichlorofluoromethane	13.3-26.7
Vinyl chloride	13.7-26.7

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Q = Concentration measured in check standard, in ug/L.

TABLE 3 - SURROGATE RECOVERY(%) CONTROL LIMITS

<u>COMPOUND</u>	<u>BLANKS</u> <u>BS, BSD</u>	<u>WATER</u> <u>MS/MSD</u>	<u>SOILS</u> <u>MS/MSD</u>
Bromochloromethane	65-135	60-140	40-130
(Trifluoromethyl)benzene	65-135	60-140	40-130

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TABLE 4 QUANTITY OF METHANOL EXTRACT REQUIRED FOR
ANALYSIS OF HIGH-LEVEL SOILS/SEDIMENTS

Approximate Concentration Range	Volume of Methanol Extract ^a
500- 10,000 ug/kg	100 uL
1,000- 20,000 ug/kg	50 uL
5,000-100,000 ug/kg	10 uL
25,000-500,000 ug/kg	100 uL of 1/50 dilution ^b

Calculate appropriate dilution factor for concentrations exceeding this table.

^aThe volume of methanol added to 5 mL of water being purged should be kept constant. Therefore, add to the 5 mL syringe whatever volume of methanol is necessary to maintain a volume of 100 uL added to the syringe.

^bDilute an aliquot of the methanol extract and then take 100 uL for analysis.

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